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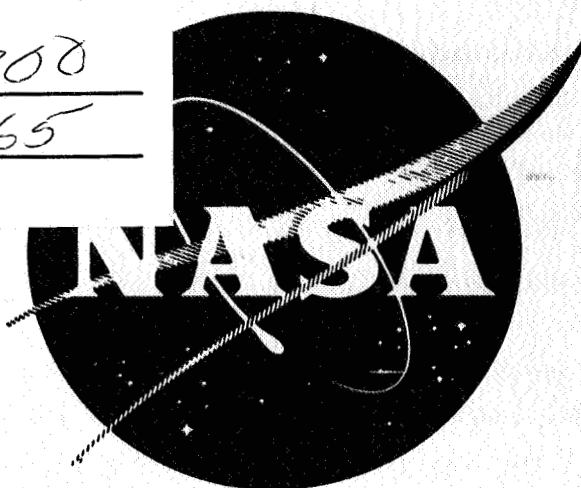
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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

Quarter Progress Report No. 10
For Quarter Ending October 15, 1967

By

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and
E. E. HOFFMAN

prepared for

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MISSILE AND SPACE DIVISION

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CINCINNATI, OHIO 45212

ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

QUARTERLY PROGRESS REPORT 10

Covering the Period
July 15, 1967 to October 15, 1967

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Under Contract NAS 3-6474

November 5, 1967

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ADVANCED REFRACTORY ALLOY CORROSION LOOP PROGRAM

I. INTRODUCTION

This report covers the period from July 15, 1967 to October 15, 1967. The primary task of this program is to fabricate, operate for 10,000 hours and evaluate a T-111 Rankine System Corrosion Test Loop. Materials for evaluation include the containment alloy, T-111 (Ta-8W-2Hf) and the turbine candidate materials Mo-TZC and Cb-132M which are located in the turbine simulator of the two-phase potassium circuit of the system. The loop design will be similar to the Cb-1Zr Rankine System Corrosion Loop; a two-phase, forced convection, potassium corrosion test loop which has been developed under Contract NAS 3-2547. Lithium will be heated by direct resistance in a primary loop. Heat rejection for condensation in the secondary potassium loop will be accomplished by radiation in a high vacuum environment to the water cooled chamber. The compatibility of the selected materials will be evaluated at conditions representative of space electric power system operating conditions, namely:

- a. Boiling temperature, 2050°F
- b. Superheat temperature, 2150°F
- c. Condensing temperature, 1400°F
- d. Subcooling temperature, 1000°F
- e. Mass flow rate, 40 lb/hr
- f. Boiler exit vapor velocity, 50 ft/sec
- g. Average heat flux in plug (0-18 inches), 240,000 BTU/hr ft²
- h. Average heat flux in boiler (0-250 inches), 23,000 BTU/hr ft²

In addition to the primary program task cited above the program also includes capsule testing to evaluate advanced tantalum alloys of the ASTAR 811 type (Ta-8W-1Hf-1Re) in both potassium and lithium.

Also included in the program is the fabrication, 5000-hour operation and evaluation of a 2600°F, high flow velocity, pumped lithium loop designed to evaluate the compatibility of the ASTAR 811 type alloys, T-111, T-222, and the tungsten alloy W-25Re-30Mo.

II. SUMMARY

The fabrication and postweld annealing of all major loop assemblies was completed.

Indicated high-oxygen concentrations in the distilled lithium by fast neutron activation analysis were partially a result of experimental technique at General Atomic. Vacuum distillation analysis has indicated an oxygen concentration of approximately 80 ppm.

The program has been modified to include the construction, operation, and evaluation of a 2600°F lithium loop.

ASTAR 811C oxygen contaminated specimens were prepared and capsules were formed for the advanced tantalum alloy capsule tests.

III. PROGRAM STATUS

A. T-111 RANKINE SYSTEM CORROSION TEST LOOP FABRICATION

The fabrication status of T-111 Corrosion Loop components is as follows:

1. Slack Diaphragm Transducers

Six T-111 alloy transducers, which were filled with NaK by Taylor Instrument Company, were received. The sampler which was filled with NaK at the time of transducer filling was received. Chemical analysis indicated less than 3 ppm oxygen concentration in the NaK. The six T-111 alloy transducers were subsequently released for loop fabrication.

2. Metering and Isolation Valves

The final machined T-111 alloy valve bodies and Mo-TZM alloy valve plugs were received. The valve plugs are shown in Figure 1. Because the valves have Cb-1Zr alloy bellows the normal welding and post-weld annealing sequence was somewhat modified. The T-111 alloy valve bodies were annealed separately at 2400°F for 1 hour per specification SPPS 03-0037-00-A. The closure weld between the valve body and bellows assemblies was then made. The completed on-off valve was annealed at 2200°F for 1 hour, the postweld heat treatment for Cb-1Zr alloy, during the postweld anneal of the potassium surge tank assembly.

The metering valve was annealed separately at 2200°F for 1 hour. A routine mass spectrometer helium leak test indicated a minute leak after this treatment. A fluorescent penetrant inspection was then performed which indicated a very slight porosity near the edge of the weld used to join the T-111 bellows assembly to the T-111 valve body. An electron beam weld pass was made over this area which eliminated the previously described leak. The valve was then postweld annealed again at 2200°F for 1 hour. No mass spectrometer leak indications were evident after this treatment, and the valve was released for incorporation into the potassium pump outlet line.

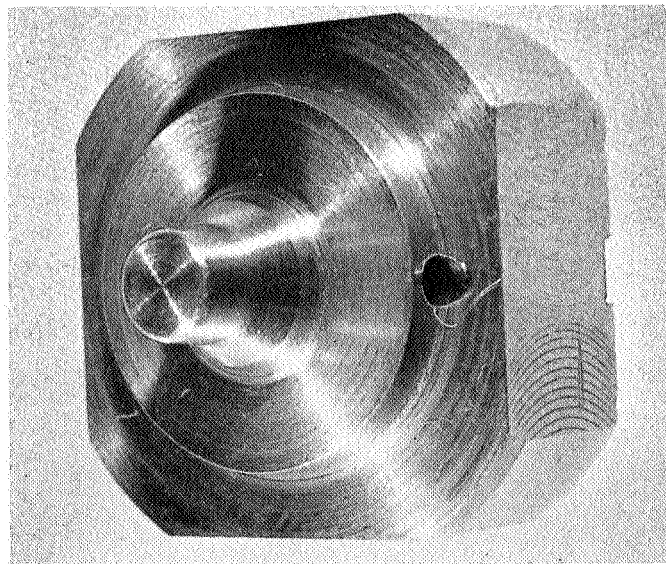
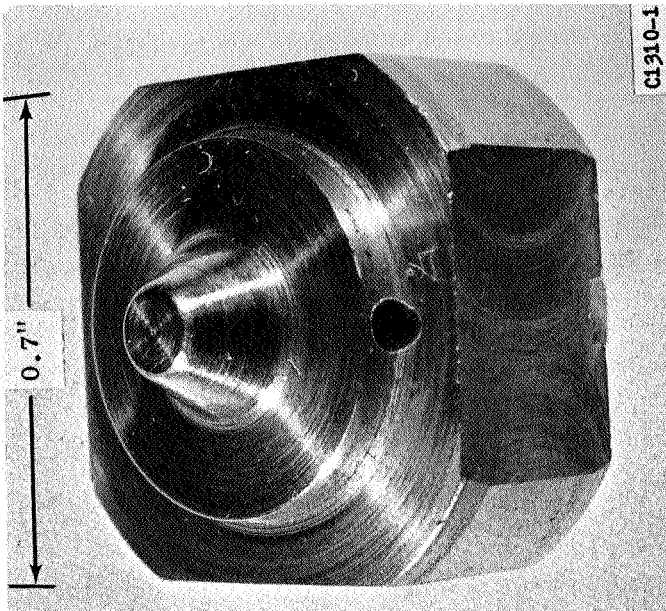


Figure 1. Mo-TZM Valve Plugs for the Metering Valve (left) and the Isolation Valve (right) of the Potassium Circuit of the T-111 Corrosion Test Loop.

(C67072443, C67072441)

3. Turbine Simulator

The single stage turbine simulator was completed and has been incorporated into the boiler assembly.

The nozzle assemblies for the nine-stage turbine simulator were also received. Each assembly consists of a nozzle, blade, and two pads which support the blade. Final hand polishing using 30 and 15 micron diamond paste was used to improve the surface finish of the nozzles. The excellent appearance of the nozzles may be seen in Figure 2. The second stage Mo-TZC alloy nozzle assembly is shown in Figure 3 and the sixth stage Cb-132M alloy nozzle assembly is shown in Figure 4. Fluorescent penetrant inspection revealed three cracked Mo-TZC alloy blade support pads in the initial group. These pads were replaced with new Cb-132M alloy pads which passed inspection. After final weighing and cleaning operations, the nozzle assemblies were positioned in the turbine simulator casings as depicted in Figure 5. A 0.062-inch diameter T-111 alloy wire is used to align the nozzle assemblies inside the casing by sliding into matching 0.032-inch grooves on the inside diameter of the casing and outside diameter of each nozzle assembly.

4. Lithium and Potassium Surge Tank Assemblies

The potassium surge tank assembly was completed with the welding of the T-111 alloy isolation valve to the Cb-1Zr tank. This assembly and the lithium surge tank assembly were then postweld annealed at 2200°F for one hour in the Union Carbide Company's ABAR furnace at Kokomo, Indiana. This anneal was conducted in accordance with specification SPPS 03-0037-00-A, except that the pressure was 5×10^{-5} torr at the start of the one hour - 2200°F run rather than the maximum of 1×10^{-5} torr specified. After 1 hour at 2200°F the pressure had decreased to 1×10^{-5} torr. The results of the chemical analysis of the test specimen which accompanied the loop components are given in Table I and indicate that no significant increase in interstitial element concentration occurred during annealing.

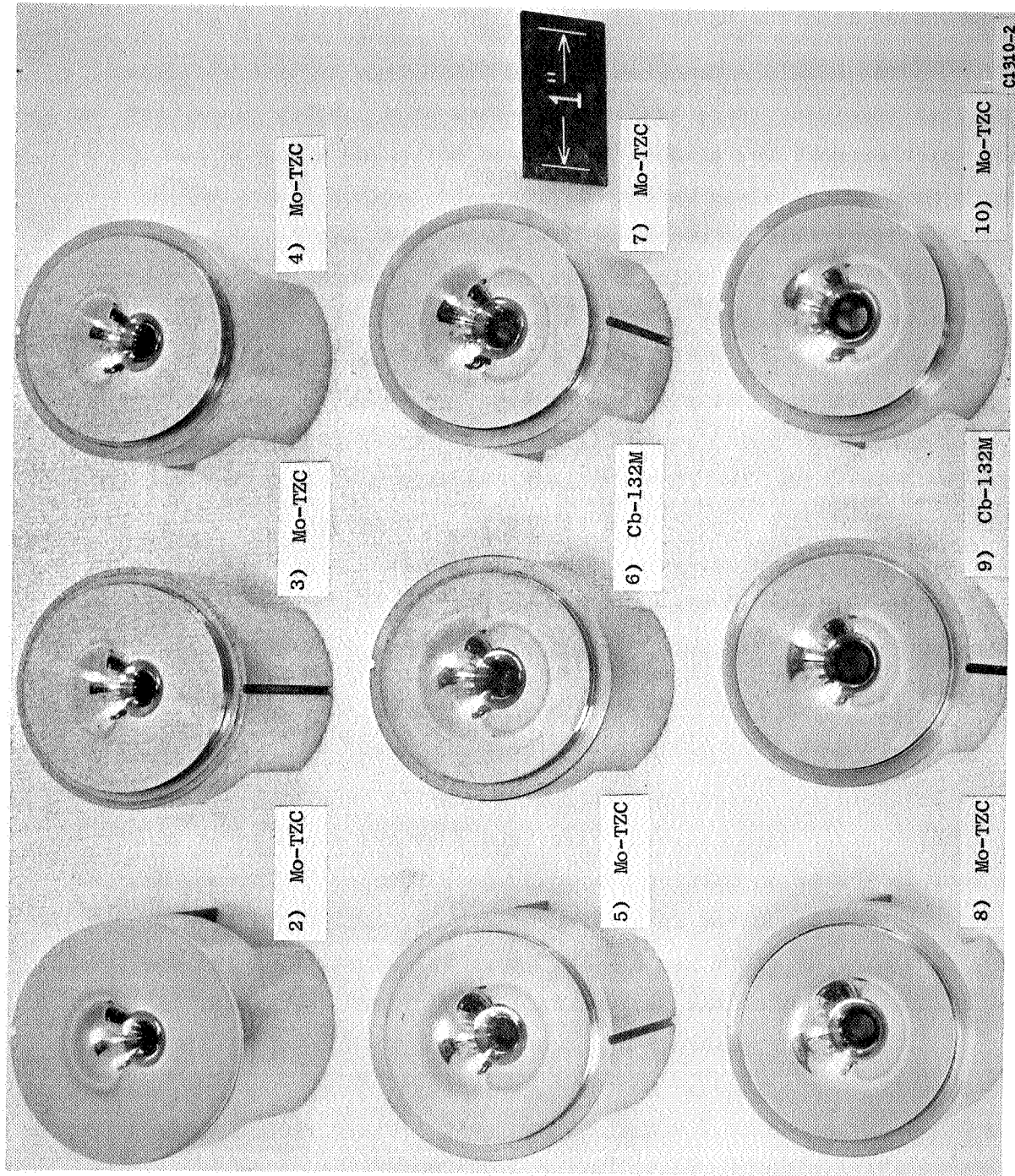


Figure 2. Test Nozzles for Turbine Simulator (Stages 2 to 10) of the T-111 Corrosion Test Loop Prior to Assembly.

(67090663)

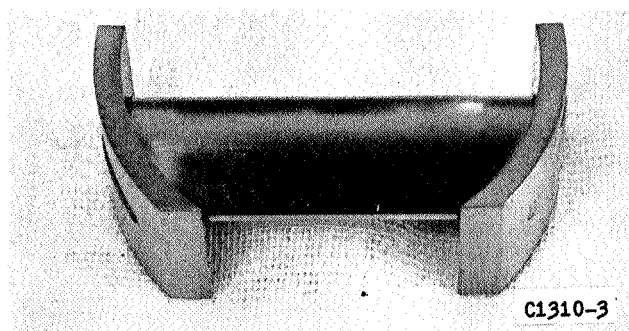
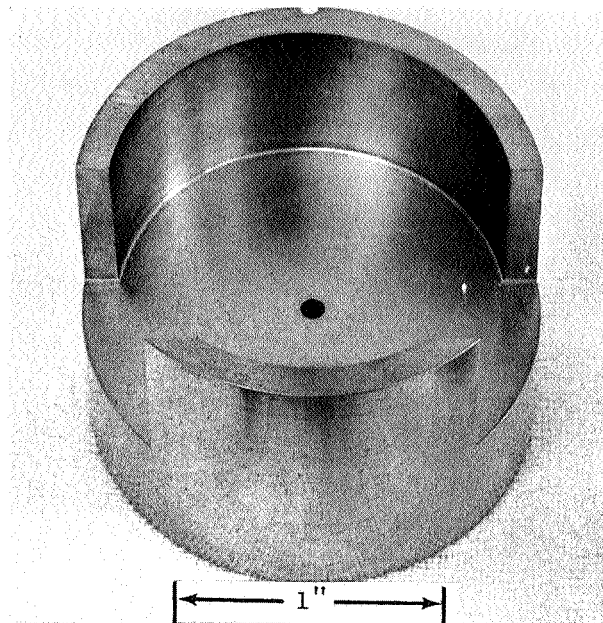
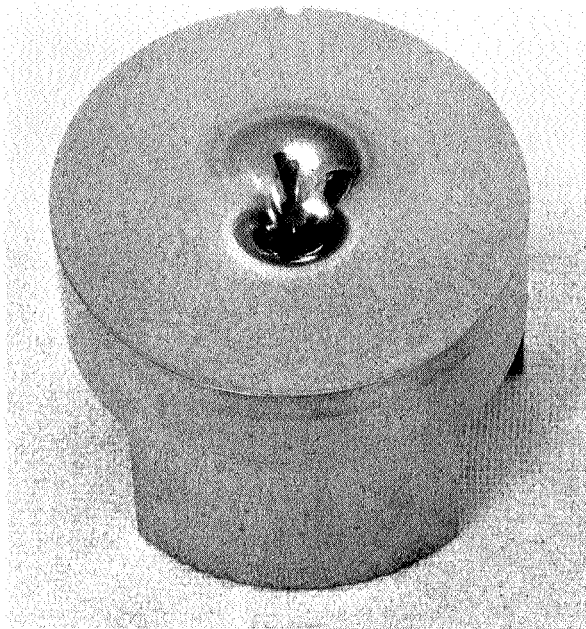


Figure 3. Entrance and Exit Side of the Nozzle and the Blade Assembly of the Second Stage (Mo-TZC) of the Turbine Simulator.
(C67090666, C6709670)

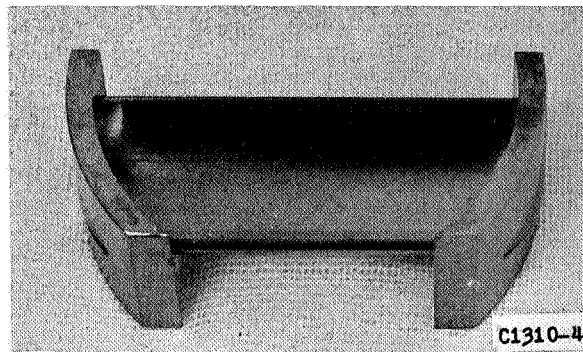
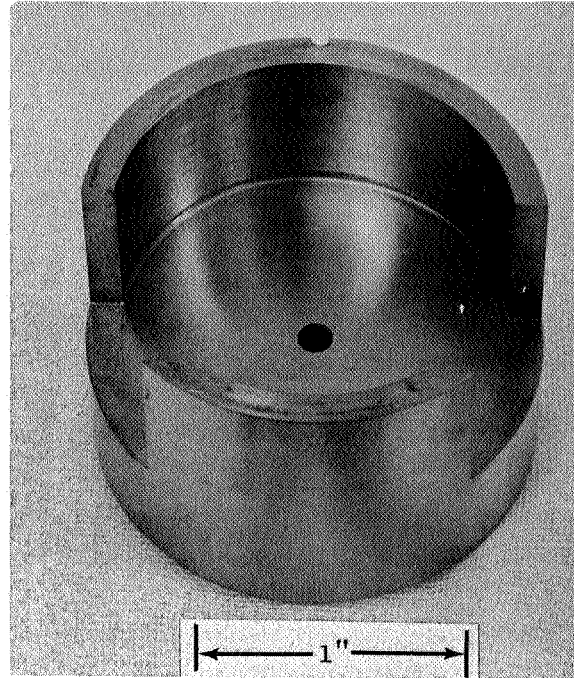
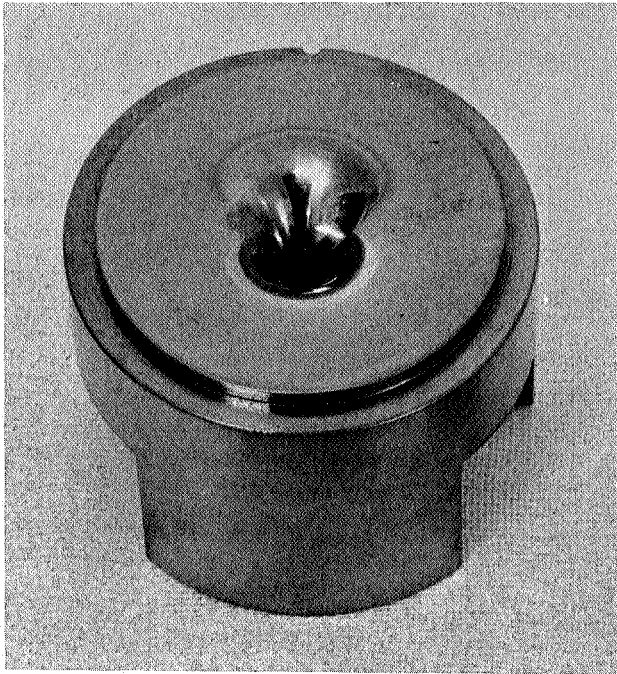


Figure 4. Entrance and Exit Side of the Nozzle and the Blade Assembly of the Sixth Stage (Cb-132M) of the Turbine Simulator.
(C67090667, C67090669)

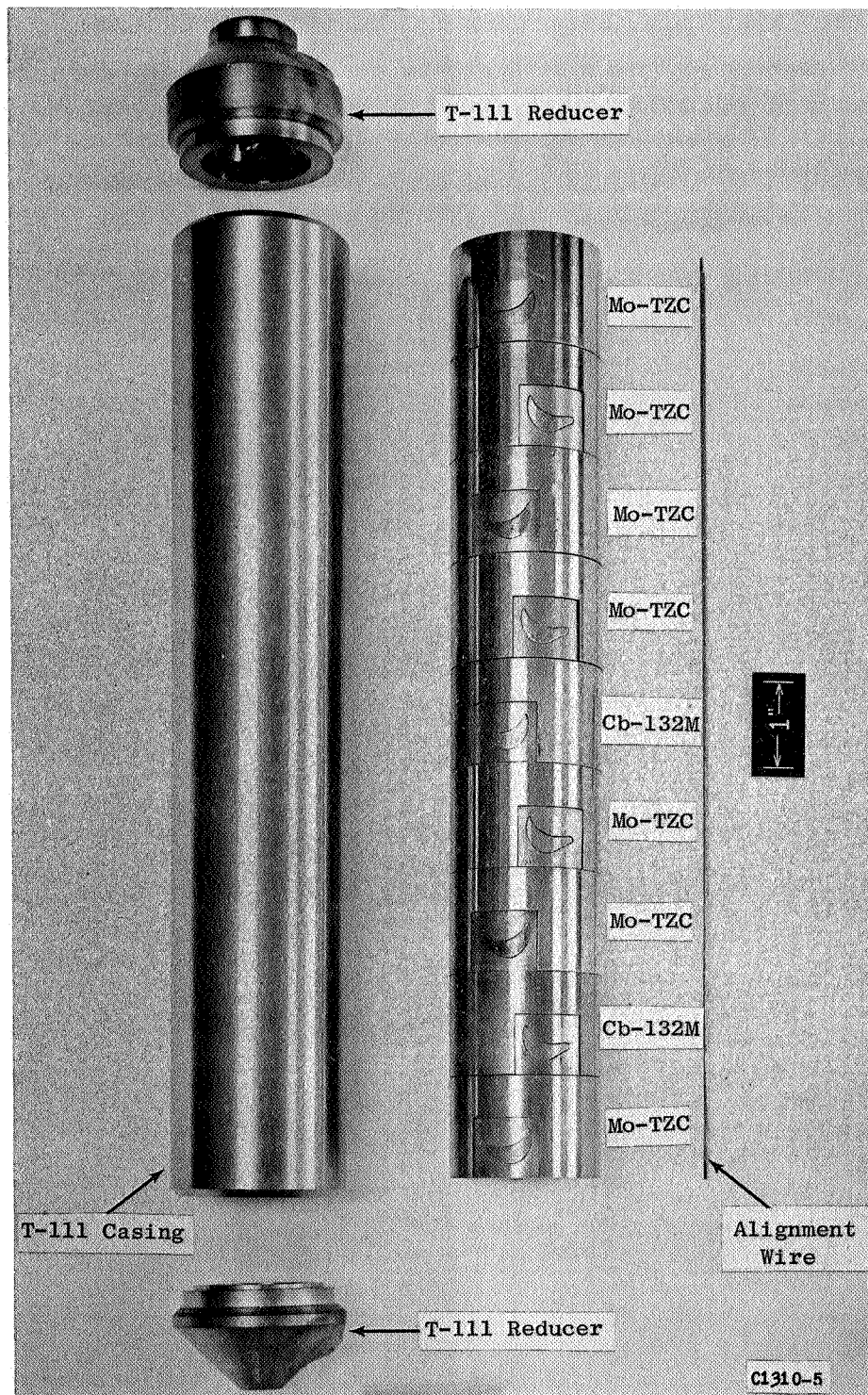


Figure 5. Turbine Simulator (Stages 2 to 10) of the T-111 Corrosion Loop I Prior to Assembly.

(C67090661)

TABLE I. RESULTS OF CHEMICAL ANALYSES OF T-111 SHEET SPECIMEN FOLLOWING
HEAT TREATMENT^(a) OF THE LITHIUM AND POTASSIUM SURGE TANK
ASSEMBLIES OF THE T-111 CORROSION TEST LOOP

	Pressure, Torr		Element, ppm ^(b)			
	Start of Run	End of Run	O	N	H	C
Pretest Analysis (MCN 02B-010) ^(c)			65	5	1	10
Analysis after one hour-2200°F vacuum anneal ^(d)	5.0×10^{-5}	1.0×10^{-5}	73 74	7 9	1 1	14 20

- (a) Vacuum heat treatment performed in Abar-90 furnace at Stellite - UCC.
- (b) Analytical methods: O, N, and H; Vacuum Fusion
C; Combustion Conductometric
- (c) 0.040-inch thick T-111 sheet
- (d) Specimen was wrapped with one layer of 0.002-inch thick Cb-1Zr foil.

Cb-1Zr to Type 316 stainless steel bimetallic joints were then welded to the gas pressurization and liquid metal inlet lines of each surge tank. These four welds were then postweld annealed locally in accordance with specification SPPS 03-0037-00-A.

5. Major Loop Subassemblies

During loop fabrication, four major subassemblies, the condenser, boiler, potassium surge tank, and lithium heater, will be sequentially assembled as described in Figure 6.

a. Condenser Assembly

The condenser assembly consists of the condenser, nine-stage turbine simulator, subcooler reservoir, and associated piping as shown in Figure 7. A more detailed view of the turbine simulator and potassium vapor line is shown in Figure 8. Radiographic inspection of this assembly revealed a 0.060-inch diameter spherical defect in the weld between the subcooler reservoir and the condenser. Because of scheduling difficulties with the large vacuum furnace required to postweld anneal this assembly, this weld repair was postponed until the assembly anneal was completed as described below. Subsequently, the defect was removed and the weld repair completed. This weld was then locally annealed at 2400°F for one hour in accordance with specification SPPS 03-0037-00-A.

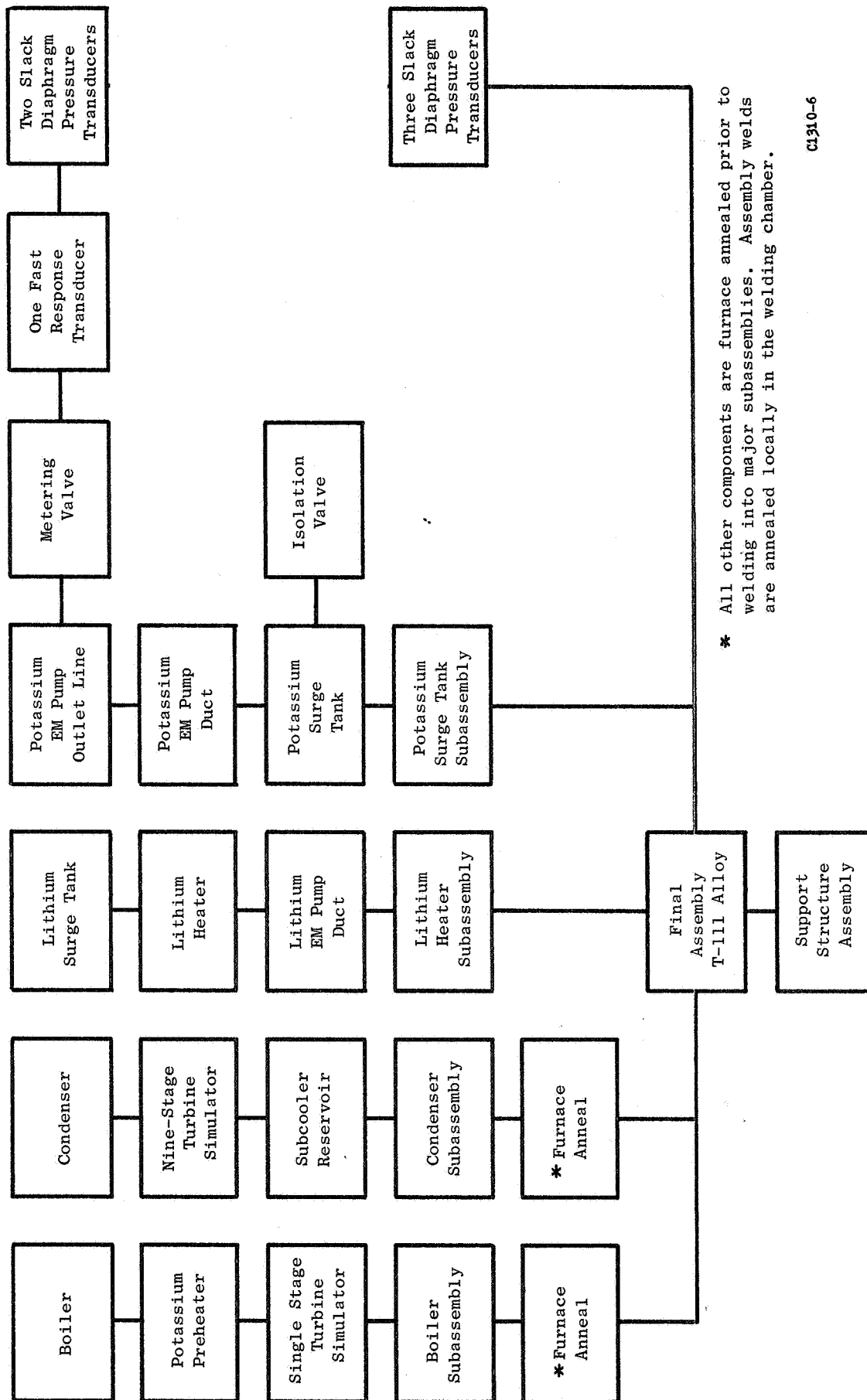
The high emittance coating of iron titanate (Fe_2TiO_5) was then applied to the unalloyed tantalum condenser fins by Pratt and Whitney Aircraft using the procedures established previously^(1,2).

b. Boiler Assembly

The boiler assembly consists of the boiler, single-stage turbine simulator, and potassium preheater as shown in Figure 9. This assembly was completed early in this report period.

(1) Potassium Corrosion Test Loop Development, Quarterly Progress Report No. 8 for Period Ending April 15, 1965, NASA Contract NAS 3-2547, NASA-CR-54735, p. 37.

(2) Recommended Procedure, December 14, 1965, Pratt and Whitney Aircraft, East Hartford, Connecticut.



CJ310-6

Figure 6. Fabrication Sequence for the T-111 Rankine System Corrosion Test Loop.

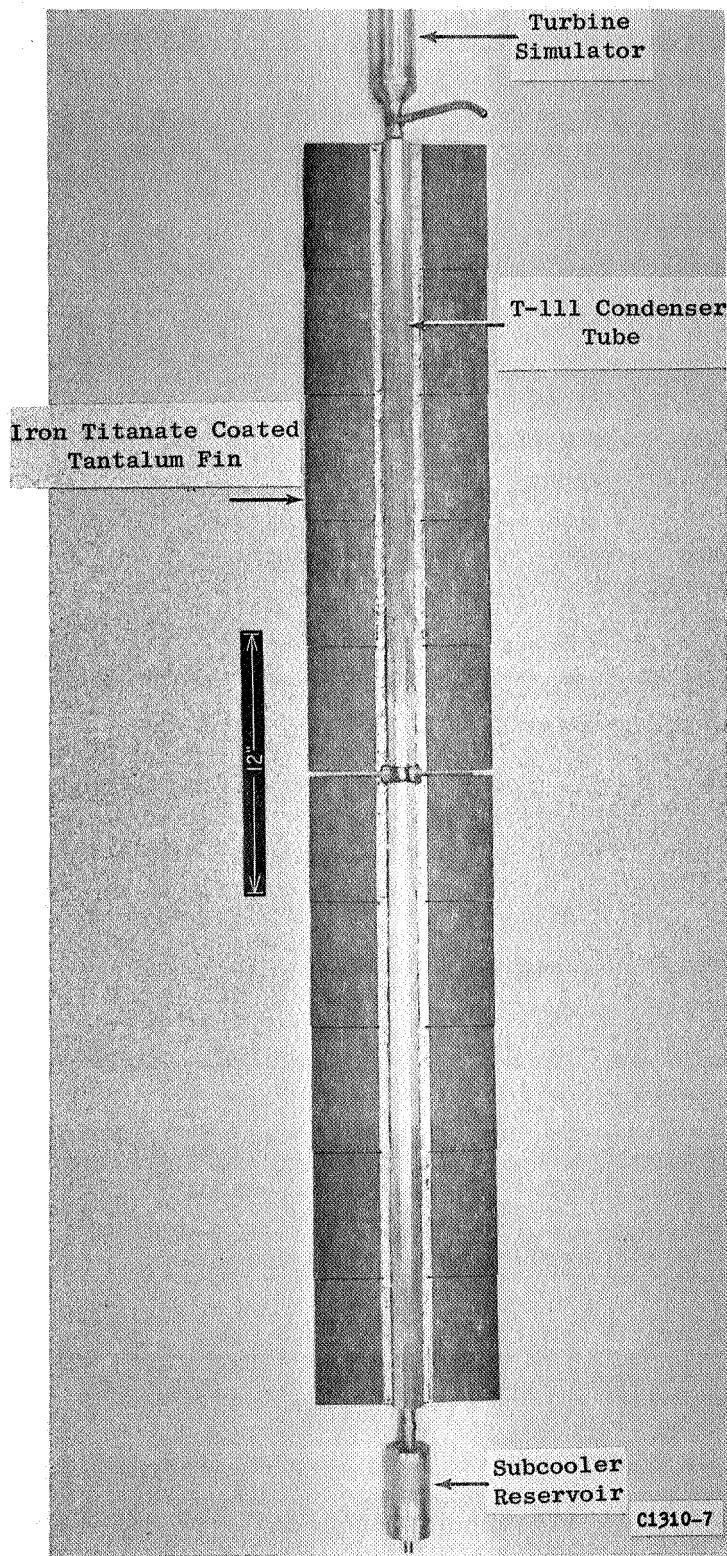


Figure 7. Condenser - Turbine Simulator Assembly Following Application of Iron Titanate Coating on Tantalum Fins.

(C67100410)

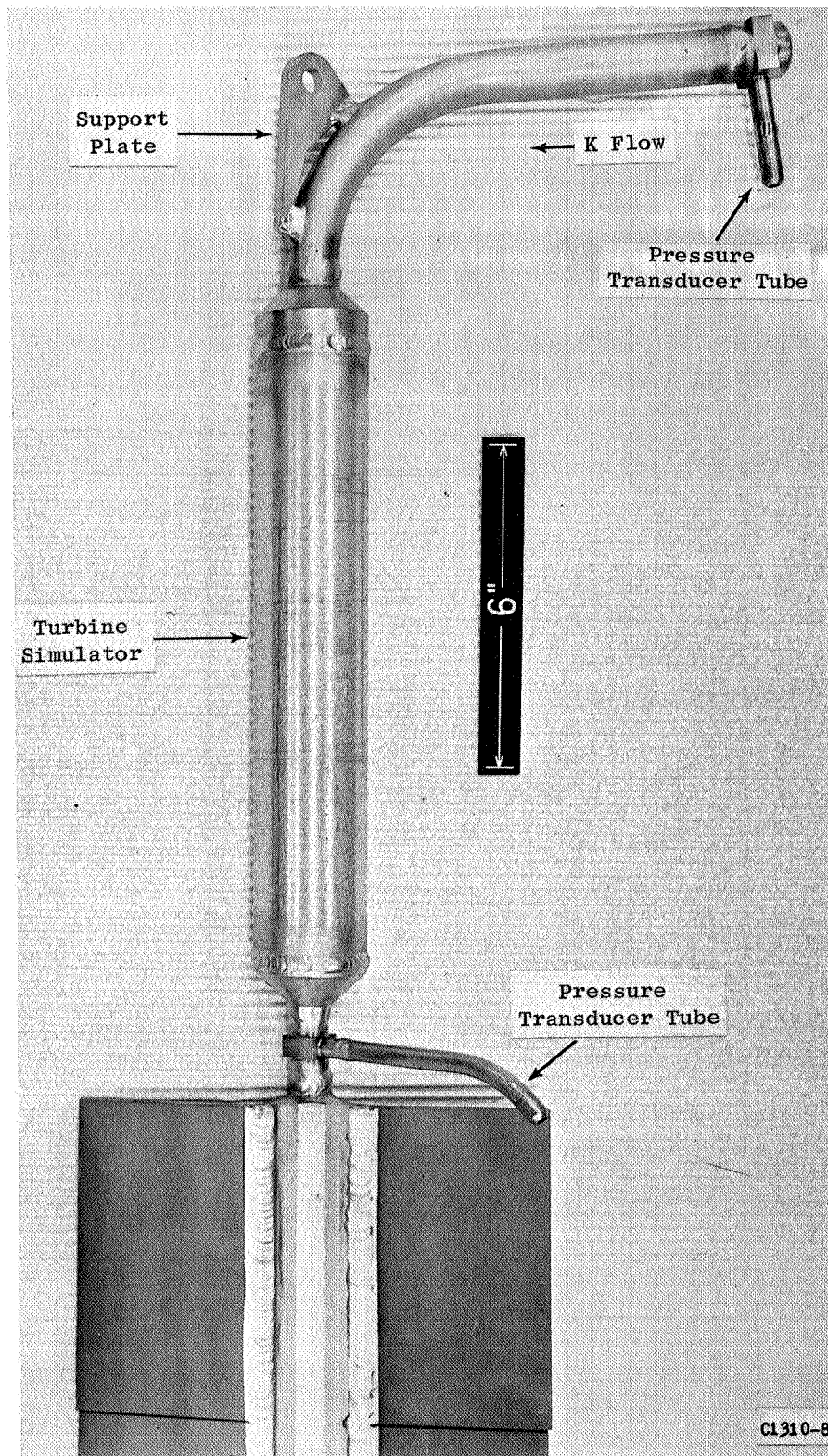


Figure 8. Nine Stage Turbine Simulator and Top of Condenser.

C67100409)

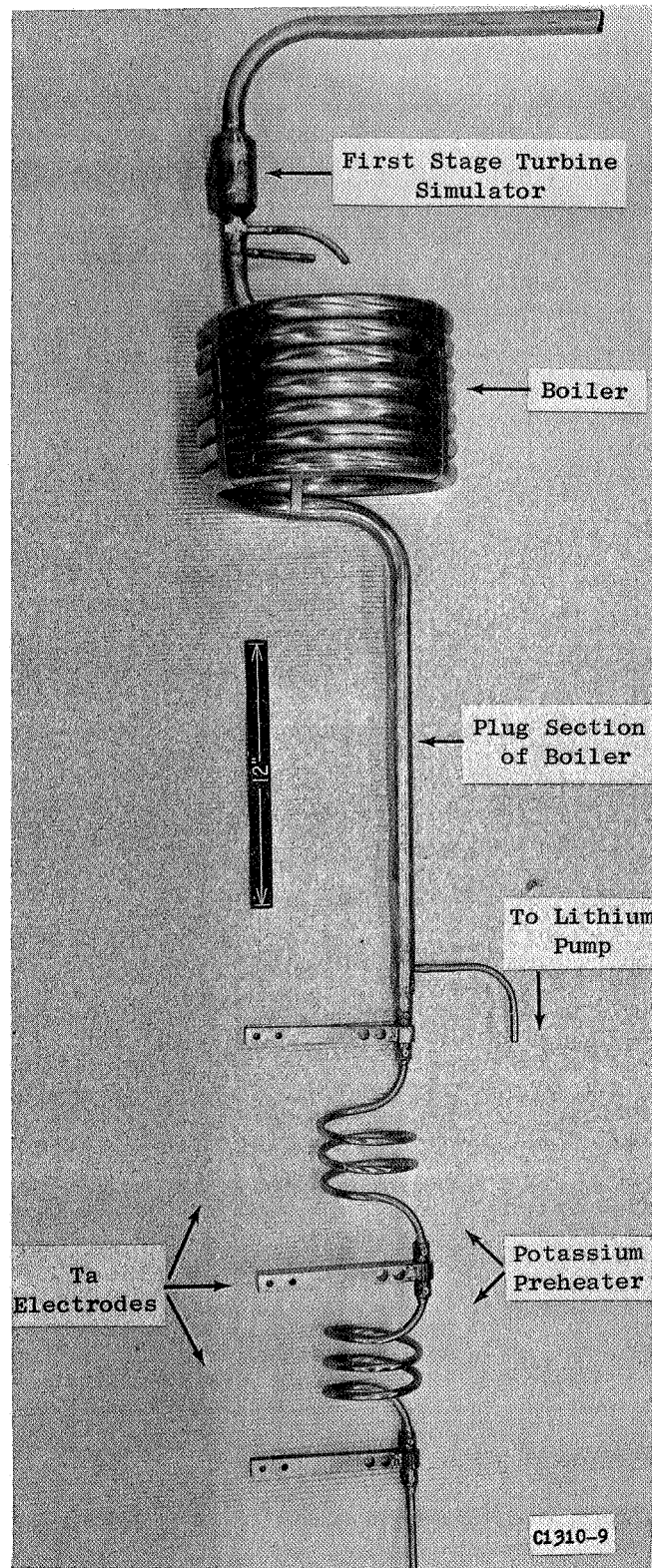


Figure 9. Potassium Preheater - Boiler - First Stage Turbine Simulator Assembly Following Postweld Anneal.

(C67100412)

c. Potassium Surge Tank Assembly

The potassium surge tank assembly consists of the potassium surge tank, potassium EM pump duct, and potassium EM pump outlet line. The outlet line contains the metering valve, two slack diaphragm pressure transducers and one fast response pressure transducer. The assembly was completed during this report period. Three local postweld anneals at 2400°F for one hour were necessary for heat treatment of nine assembly welds.

d. Lithium Heater Assembly

Three welds will be required to join the lithium heater, EM pump duct, and surge tank of the lithium heater assembly. These welds and the two local postweld anneals required will be performed early in the next report period.

6. Postweld Annealing of Major Loop Subassemblies

The boiler and condenser major subassemblies and the lithium heater, all wrapped with one overlapping layer of Cb-1Zr foil, were postweld annealed at 2400°F for one hour in Stellite's Brew furnace Model 966 at Kokomo, Indiana on September 7, 1967. This anneal was conducted in accordance with specification SPPS 03-0037-00-A. This furnace had previously been qualified at 3000°F using 0.040-inch thick T-111 alloy test coupons both wrapped with Cb-1Zr alloy foil and unwrapped. The results of this qualification run are shown in Table II and indicate no significant increase in interstitial element concentration occurred during annealing.

During the annealing the various loop assemblies, which weighed approximately 250 pounds, were suspended from Ta-10W alloy hanger bars that are mounted on the top flanged dome of the chamber.

Four Pt/Pt-10Rh thermocouples were used to monitor the temperature of the loop components. These thermocouples were wrapped with two layers of Cb-1Zr foil and positioned under the Cb-1Zr foil wrap which surrounded the components. A W-5Re/W-26Re thermocouple was used to monitor the furnace temperature.

TABLE II RESULTS OF THE QUALIFICATION TEST OF STELLITE'S BREW FURNACE
MODEL 966 ON AUGUST 25, 1967

Furnace Conditions: 1 Hour at 3000°F
Maximum Pressure at Temperature: 4.4×10^{-5} Torr

Element ^(a)	Concentration, ppm ^(b)		
	Before Anneal	Wrapped Specimen ^(c) After Anneal	Unwrapped Specimen After Anneal
O	26	27	51
N	14	12	14
H	1	1	2
C	<10	11	27

- (a) Test specimens: T-111 sheet, 0.040-inch thick (MCN 02A-078)
- (b) Analytical methods: O, N, and H; Vacuum Fusion
C; Combustion Conductometric
- (c) Specimen was wrapped with layer of 0.002-inch thick Cb-1Zr foil during heat treatment.

The furnace temperature and pressure during the annealing run are given in Figure 10. The furnace heat up rate, which was programmed at 25°F/minute, was interrupted and the temperature held constant at 1000°, 1500°, 2040°, and 2200°F for time periods of 10 to 15 minutes to allow time for thermal equilibration, outgassing and pressure reduction. The beneficial effect of this holding operation on outgassing of the system is evident in Figure 10. The chamber pressure was 7×10^{-6} torr at the start of the one hour anneal and decreased to 3×10^{-6} torr after one hour. The temperature of the various components and the correlation of these temperatures with furnace temperature and chamber pressure are given in Table III. It may be noted that the temperatures of the various components equilibrated with the furnace temperature in less than 10 minutes at 2400°F. The maximum temperature difference for the three components was 25°F, indicated excellent temperature uniformity within the furnace.

B. ALKALI METAL PURIFICATION

Due to the fact that recent analyses of distilled lithium for oxygen by fast neutron activation indicated high values, 245 ppm and 138 ppm⁽³⁾, another sample was removed from the still receiver. This lithium sample was submitted to General Atomic for analysis along with a sample of potassium known to contain less than 10 ppm oxygen. The results, 800 ppm \pm 65 ppm oxygen for the lithium and 485 ppm \pm 44 ppm oxygen for the potassium, indicated that these samples were contaminated during the capsule loading operation at General Atomic. Consequently, polyethylene capsules were obtained from General Atomic and loaded with distilled lithium and low-oxygen potassium at SPPS in the Vasco welding chamber in a helium atmosphere which contained the following contaminants in ppm as determined by analysis with a mass spectrometer and electrolytic hygrometer:

	<u>O</u>	<u>N</u>	<u>H₂O</u>
Before loading	2	2	0.3
After loading	2	2	2.3

(3) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report Number 9 for Period Ending August 15, 1967, NASA Contract NAS 3-6474.

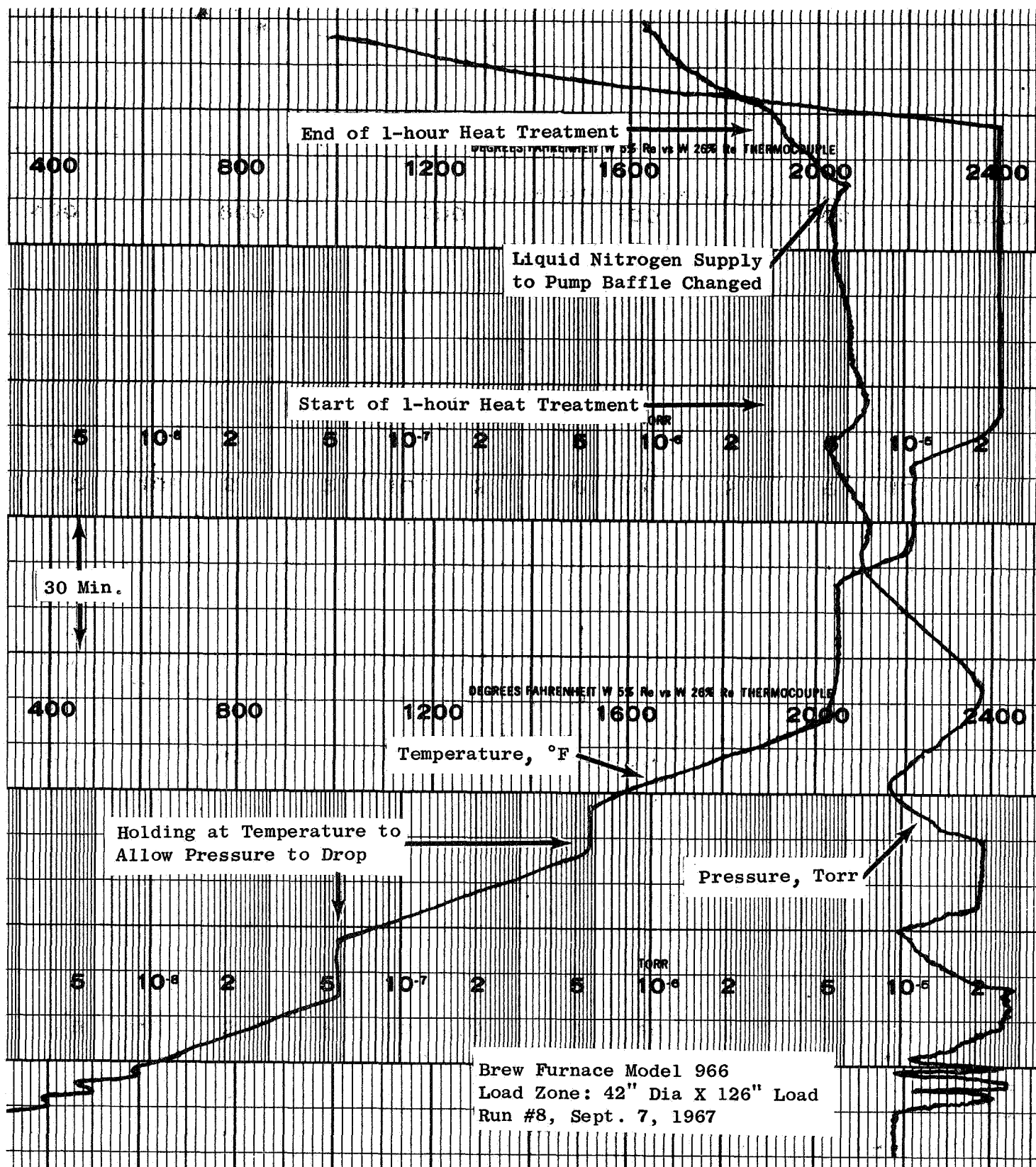


Figure 10. Pressure and Temperature During the Heat Treatment of Corrosion Loop I Components in the Brew Furnace at Stellite on September 7, 1967. C1310-10

TABLE III TEMPERATURES AND PRESSURE DURING 2400°F-1 HOUR ANNEAL OF T-111 CORROSION TEST LOOP ASSEMBLIES
IN STELLITE'S BREW FURNACE MODEL 966 ON SEPTEMBER 7, 1967

Time	Pressure, Torr	Temperature, °F				Remarks
		Furnace	T/C-1 (a)	T/C-2 (b)	T/C-3 (c)	T/C-4 (d)
1409	3.0×10^{-5}	1000	840	840	750	710
1420	1.0×10^{-5}	1000	860	885	800	780
1440	2.0×10^{-5}	1500	1320	1380	1190	1290
1450	9.0×10^{-6}	1510	1380	1470	1290	1420
1510	2.0×10^{-5}	2020	1920	1970	1970	1920
1520	1.6×10^{-5}	2040	2000	2015	2000	2000
1530	1.0×10^{-5}	2040	2010	2020	2010	2010
1540	7.0×10^{-6}	2040	2010	2025	2030	2030
1547	7.0×10^{-6}	2200	--	--	--	--
1600	6.0×10^{-6}	2220	2175	2190	2180	2175
1620	7.0×10^{-6}	2400	2360	2390	2365	2365
1630	6.0×10^{-6}	2410	2375	2400	2390	2390
1700	5.0×10^{-6}	2410	2375	2400	2390	2390
1725	3.0×10^{-6}	2410	2375	2400	2390	2390
1735	1.6×10^{-6}	1500	--	--	--	--

- (a) T/C-1: Inside boiler coil, 3" from top
(b) T/C-2: Boiler plug, 5" from bottom
(c) T/C-3: Condenser, 5" from top
(d) T/C-4: Lithium heater, middle electrode

The length of time during which the alkali metal specimens were exposed to the chamber atmosphere was less than 30 minutes. The polyethylene capsules were sealed by melting the polyethylene with a soldering gun using a new copper tip. The capsules were subsequently sealed in stainless steel tubes with Swagelok caps while still in the welding chamber. The alkali metal surface showed no visible signs of oxygen contamination at the time of sealing. Fast neutron activation analysis of these specimens for oxygen indicated 1530 ± 133 ppm and 1473 ± 132 ppm for the lithium and 814 ± 70 and 867 ± 71 ppm for the potassium. General Atomic was requested to investigate all possible sources of error to determine the reason for these high values. They discovered that the polyethylene capsules used in recent analyses were from a new lot for which the average oxygen blank was considerably higher than that for the previous lot which was used in the calculations. The average blank for the new lot was then determined, and values for oxygen of 461 ± 219 ppm and 489 ± 217 ppm for the lithium, and 215 ± 114 ppm and 229 ± 114 ppm for the potassium were found. These results are still considered to be exceptionally high. It must be emphasized that the capsule blank used in the calculations was an average value, and the range of values from the capsules which gave this average is not known. Future neutron activation analyses at General Atomic will be performed using capsules for which the oxygen blank has been determined on an individual basis.

A sample of the lithium, for which the exceptionally high oxygen values were obtained by neutron activation, was also sent to Mr. Randolph Gahn at the Lewis Research Center (NASA) for analysis by the distillation method being developed there. He obtained values of 83 and 87 ppm oxygen. No analytical blank has been subtracted from these values since the blank for this system has not been determined. The true oxygen values may therefore be even less than indicated. In addition, two samples of this lithium, of equal weight, were spiked with oxygen. The average oxygen concentration of the lithium was calculated from the previous analyses and indicated that prior to spiking the samples contained $26 \frac{1}{2}$ gm oxygen. One sample was

spiked with 142 μ gm oxygen and the other, with 284 μ gm oxygen; the recoveries were 85% and 96%, respectively.

Sufficient lithium to fill the T-111 Corrosion Test Loop and the 2600°F Lithium Loop shall be distilled prior to obtaining additional oxygen determinations.

C. 2600°F LITHIUM LOOP

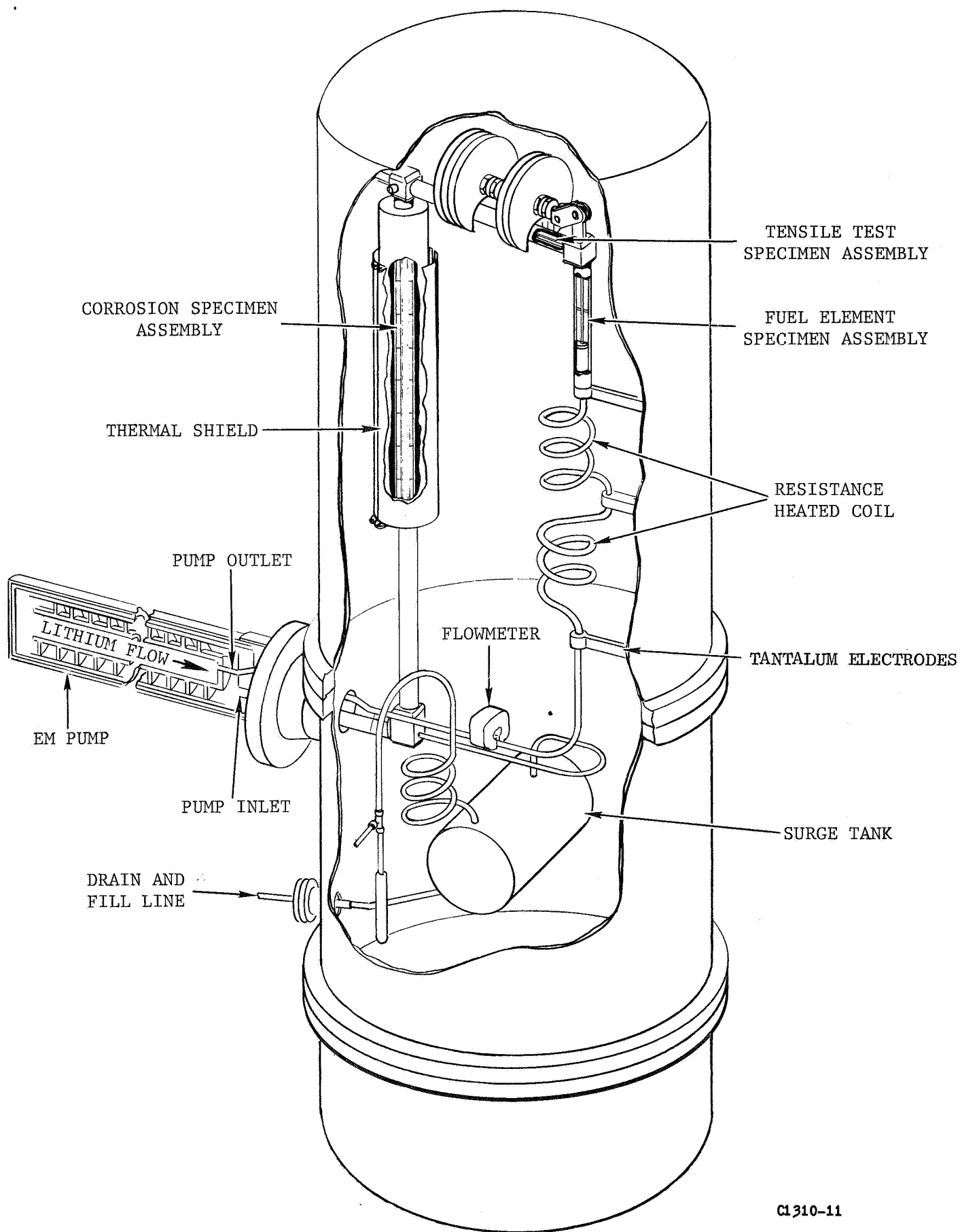
The Advanced Refractory Alloy Corrosion Loop Program has been modified to include the design, fabrication, instrumentation, operation and evaluation of a 2600°F Lithium Loop. The primary objective of the test program is to determine the corrosion resistance of several advanced refractory alloys to liquid lithium in a test loop which simulates the maximum temperature, temperature gradient, heat flux and lithium velocity of proposed out-of-pile thermionic power generation systems. The loop will be fabricated of T-111 alloy and contain advanced refractory alloy specimens. The loop will be operated for 5,000 hours at a maximum temperature of 2600°F with a temperature gradient of 200°F in the heat rejection section of the loop.

The detail design of the 2600°F Lithium Loop was completed and submitted to the NASA Program Manager for approval. The loop is illustrated in an isometric drawing, Figure 11 which shows the relative position and orientation of the principle loop components. A schematic diagram of the loop at steady state operating conditions is shown in Figure 12.

The principle components of the loop and their functions are described below:

1. Electromagnetic Pump

A helical induction electromagnetic pump with a T-111 alloy duct will circulate liquid lithium at a flow rate of 400 pounds per hour at an inlet temperature of 2400°F. The duct will be wrapped with Cb-12r foil insulation and enclosed in a stainless steel can which will be welded to a 4-inch diameter port on the vacuum chamber completing the vacuum tight enclosure around the T-111 alloy duct. The pump stator is mounted



CI310-11

Figure 11. Isometric Drawing of 2600°F Lithium Loop.

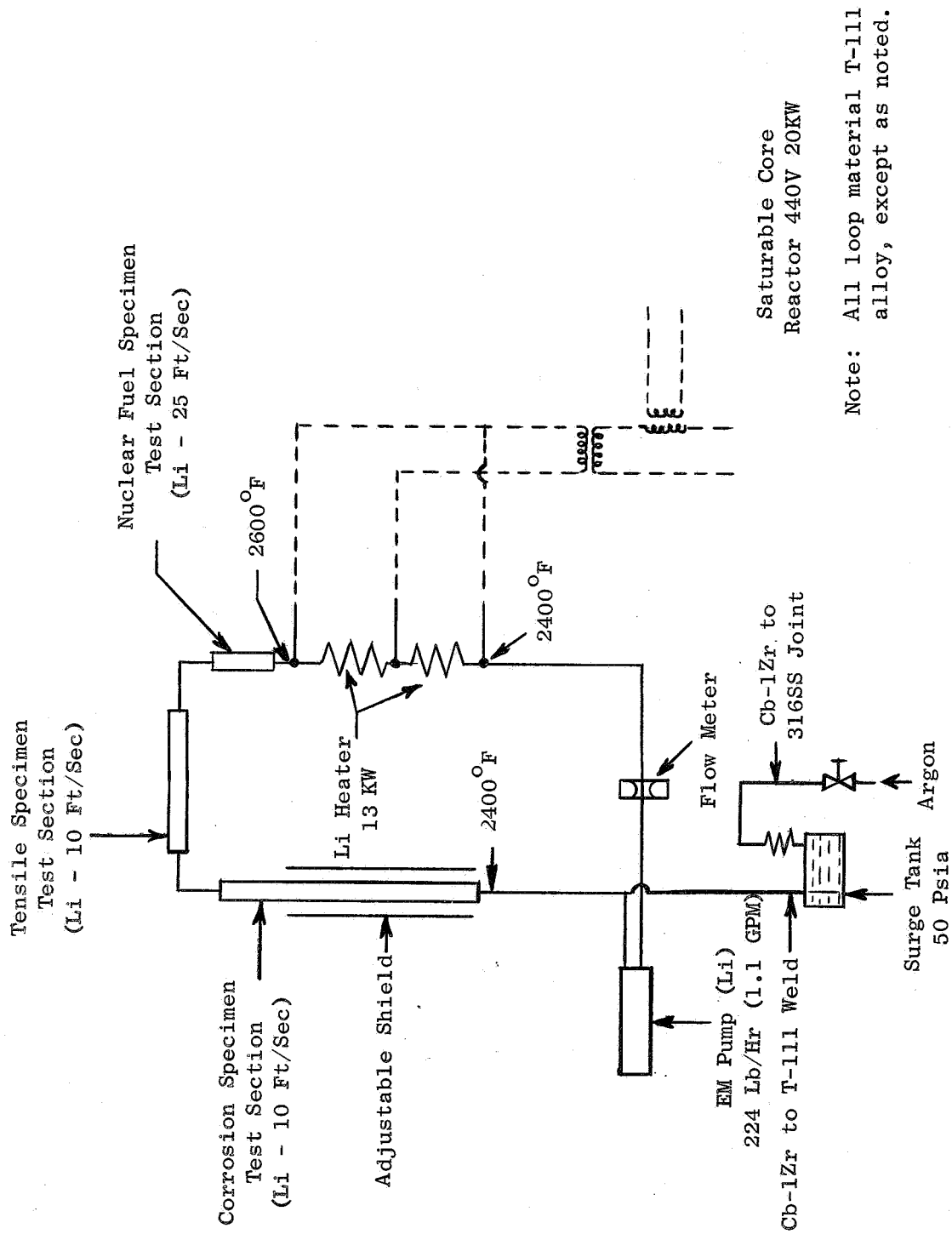


Figure 12. Schematic Diagram of 2600°F Lithium Loop. C1310-12

on an overhead trolley that allows it to slide over the stainless steel can. The electrical windings of the stator are air-cooled by a blower mounted on the stator casing.

2. Electrical Resistance Heater

The electrical resistance lithium heater consists of two sections of 0.375-inch OD by 0.065-inch wall T-111 tubing wound in a 4-inch diameter coil. At each end of the heater and at the center, a 1-inch diameter section will be butt welded to the tubes as a support and for the attachment of the electrodes. The electrodes which are not in contact with lithium are made from unalloyed tantalum. A 20KW saturable core reactor with a high current, step down transformer will be used to supply electrical power to the lithium heater. The heater is designed to operate at lithium exit temperatures of 2600°F in steady state operation.

A 0.125-inch diameter, T-111 alloy rod insert will be welded in each heater coil to increase the local lithium velocity to 10 feet per second and supply specimens for post-test evaluation of the effects of high velocity liquid lithium on the T-111 alloy.

3. Nuclear Fuel Specimen Test Section

A section of the loop at the exit of the lithium heater is designed to contain three nuclear fuel specimens in series. The fuel specimens are tubular in shape with a 0.5-inch OD by 0.135-inch ID and are 1.7 inches long. The lithium in contact with the ID of each fuel specimen will have a velocity of 25 feet per second whereas stagnant lithium will be in contact with the OD of each specimen. The fuel specimen section will be wrapped with multiple layers of Cb-1Zr foil to achieve a near isothermal condition at 2600°F.

4. Tensile Specimen Test Section

A section in the top horizontal leg of the loop is designed to contain tensile specimens of three advanced refractory alloys: ASTAR 811C, ASTAR 811CN and W-25Re-30Mo. The tensile specimens are inserted in pairs

with a small gap separating each pair. In this manner the lithium velocity through the gap will be 10 feet per second with stagnant lithium in contact with the other surface of the test specimen. Identical control specimens to those in contact with lithium will be attached to the outer surface of the test section. The entire section, including the control specimens will be wrapped with multiple layers of Cb-1Zr foil to achieve a near isothermal condition at 2600°F. The control specimens will be evaluated in conjunction with the specimens in contact with lithium after testing to separate the thermal effects from the effects of the alkali metal exposure.

5. Corrosion Specimen Test Section

A section of the loop is designed to contain tubular specimens of three refractory alloys, T-111, ASTAR 811C and ASTAR 811CN for post-test corrosion evaluation in a 1-inch diameter container tube. A 0.375-inch tube will be inserted in the center hole of each corrosion specimen to increase the lithium velocity to 10 feet per second. The corrosion specimen test section also functions as a heat rejector for the loop, and its effective radiating area will be increased by enclosing the 1-inch diameter container tube in a 3-inch diameter lithium filled jacket that is open at the bottom to the main loop. A thermal shield will surround the assembly which can be manually adjusted to regulate the radiation heat loss to maintain a 200°F temperature gradient in the loop.

6. Electromagnetic Flowmeter

A permanent magnet flowmeter with a flux density of 3000 gauss will be used to measure the lithium flow rate. The flow tube is a 0.375-inch OD by 0.065-inch wall T-111 alloy tube. T-111 alloy wire electrodes will be welded to the tubes at right angles to the magnetic flux field. The tube and the magnet will be partially shielded by multiple layers of Cb-1Zr foil to limit the temperature of the magnet to less than 900°F during operation.

7. Test Chamber

The entire loop will be contained in a 24-inch diameter vacuum chamber capable of a cold wall vacuum of 1×10^{-9} torr. During peak outgassing loads at the start of the test, two titanium sublimation pumps will assist the 100 liter per second getterion pump to maintain the pressure in the 10^{-8} torr range. The entire loop will be supported by a stainless steel structure which will be welded to a 24-inch high center spool section which is employed to facilitate both the manufacturing and installation of the loop.

D. ADVANCED TANTALUM ALLOY CAPSULE TESTS

The ASTAR 811C and ASTAR 811CN sheet materials necessary for capsule fabrication were vacuum annealed for 1 hour at 3000°F at National Research Corporation, Newton, Massachusetts. The chemical analysis of these materials following annealing is shown in Table IV.

The ASTAR 811C and ASTAR 811CN annealed sheet materials were successfully formed into capsules at National Research Corporation on September 22, 1967. Seam welding of the capsules was performed at GE-SPPS according to specification SPPS 03-0025-00-A. A typical capsule shell and inner tube assembly for a lithium thermal convection capsule are shown in Figure 13.

Oxygen contaminated ASTAR 811C specimens were prepared in the 24-inch diameter by 54-inch high Varian high vacuum system⁽⁴⁾. The 0.040-inch thick specimens measuring 1 inch wide x 22 inches long were heated by direct resistance, and the temperature was monitored with W-3Re/W-25Re thermocouples. The chamber was initially evacuated by means of a 260 liter/sec turbomolecular pump. Following bakeout of the system, a cold wall pressure of 1.5×10^{-9} torr was achieved. The specimens were heated to 2400°F and held at a 1×10^{-6} torr pressure maintained by means of an oxygen leak. The oxygen partial pressures were determined during the run with a

(4) Advanced Refractory Alloy Corrosion Loop Program, Quarterly Progress Report Number 6, for Period Ending October 15, 1966, NASA Contract NAS 3-6474, NASA-CR-72177.

TABLE IV CHEMICAL ANALYSIS OF ASTAR 811C AND ASTAR 811CN SHEET^(a)

Element	Chemical Analysis, ppm ^(b)	
	ASTAR 811C	ASTAR 811CN
O	43, 59 ^(b)	91, 87
N	9, 8	113, 101
H	1, 1	1<1
C	290, 332	152, 174

(a) After recrystallization annealing for 1 hour at 3000°F.

(b) Analytical Methods: O, N, and H; Vacuum Fusion
C; Combustion Conductometric.

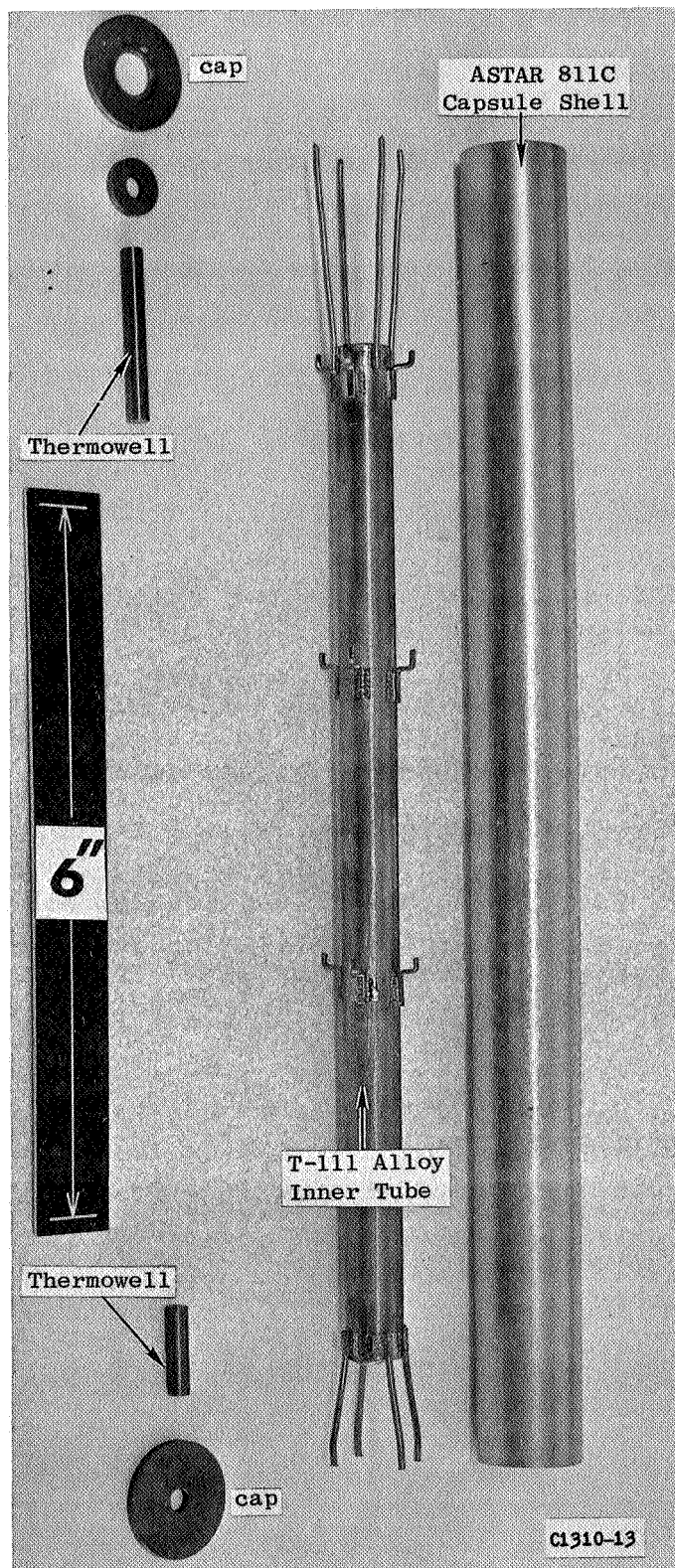


Figure 13. Lithium Thermal Convection Capsule Assembly
(ORIG. C67062922)

General Electric partial pressure gas analyzer resulting in the data shown in Table V. The chemical analysis of ASTAR 811C before and after contamination, shown in Table VI, indicates no significant changes in nitrogen, hydrogen, and carbon concentrations as a result of oxygen contamination.

TABLE V OXYGEN CONTAMINATION OF ASTAR 811C^(a) AT 2400°F IN A
 1×10^{-6} TORR PRESSURE MAINTAINED BY AN OXYGEN LEAK

Oxygen Partial Pressure, Torr ^(b)	5.7×10^{-7}
Exposure Time, Hours	11.5
Torr Hours	65.5×10^{-7}
Initial Oxygen Concentration, ppm	50
Final Oxygen Concentration, ppm	255
Oxygen Pickup, ppm	205
Oxygen Contamination Rate ppm/torr hour	31.4×10^6
Sticking Factor	.46

(a) 0.040-inch thick sheet.

(b) Average of 11 partial pressure scans ranging from 5.4×10^{-7} torr to 5.9×10^{-7} torr.

TABLE VI CHEMICAL ANALYSIS OF ASTAR 811C BEFORE AND AFTER
OXYGEN CONTAMINATION^(a)

<u>Element</u>	Chemical Analysis, ppm ^(b)	
	<u>Before Contamination</u>	<u>After Contamination</u>
O	43, 59	252, 256
N	9, 8	4, 9
H	1, 1	1, 1
C	290, 332	269, 314

(a) 11 hours at 2400°F at 1×10^{-6} torr pressure maintained by an oxygen leak.

(b) Analytical methods: O, N, and H; Vacuum Fusion
C; Combustion Conductometric.

IV. FUTURE PLANS

- A. The T-111 Rankine System Corrosion Test Loop assembly will be completed with the following major events.
 - 1. Completion of the lithium heater assembly.
 - 2. Assembly of all major subassembly in the loop support structure and final welding fixtures.
 - 3. Final loop assembly which requires eight welds to join the major subassemblies and attach three pressure transducers.
 - 4. Final assembly in the vacuum chamber spool section and permanent support structure.
- B. The lithium necessary for filling both the T-111 Corrosion Test Loop and the 2600°F Lithium Loop will be distilled and analyzed.
- C. Fabrication of 2600°F Lithium Loop components will proceed.
- D. Assembly of the advanced tantalum alloy capsules will be completed and the capsules will be filled with alkali metal.

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